

PATENT SPECIFICATION

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 (72) Inventor IVOR EDISON DENTON



(54) IMPROVEMENTS IN OR RELATING TO FOAMS

(71) We, UNITED KINGDOM ATOMIC ENERGY AUTHORITY, London, a British Authority, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of preparing a foamed, inorganic refractory material.

Foamed refractory materials may be made, for example, by mixing foamable resin precursors, a blowing agent and a refractory filler to form a green foam followed by debonding and firing. The foamable precursors may, for example, be toluene di-isocyanate and a polyol which condense to give a polyurethane, the heat generated during the condensation causing the blowing agent to volatilise or decompose to gaseous products thereby to generate the foam.

The present invention provides a method of preparing a foamed, inorganic refractory material comprising the steps of

- (i) preparing a mixture of powdered, inorganic refractory material, as defined herein, and a binder under conditions such that gas or vapour is entrapped within the mixtures;
- (ii) subjecting the mixture to sub-atmospheric pressure to foam the mixture thereby to produce a green foam; and
- (iii) subsequently debonding to remove the binder and firing to produce the final foamed inorganic refractory material.

The present method has the particular advantage that foamable resin precursors are not required. We believe that the foaming arises from the outgassing effect of the sub-atmospheric pressure.

In step (i), the refractory material and the binder may, for example, be mixed and then mechanically agitated in air so that gas (in this case air) is entrapped within the mixture.

Examples of binders which may be used in the present method are cold setting resins

such as epoxy resins, phenolic resins, polyester resins, silicone resins and polyamide resins.

The term "refractory material" as used in step (i) is to be taken to cover not only a conventionally termed refractory material but also a material which is converted to a conventionally termed refractory material in the firing of step (iii). Thus, the refractory material in step (i) is not necessarily the same as the final refractory material produced by the present method. The refractory material used in step (i) may be a refractory glass, such as petalite or a borosilicate or lithium aluminosilicate glass; a technical ceramic, such as porcelain or a lithium aluminosilicate; a pure oxide ceramic, such as alumina or zirconia; a carbide ceramic; a metal, such as stainless steel; or silicon, when the firing may be carried out in a nitriding atmosphere to convert the silicon to silicon nitride.

In step (ii), we prefer to use a sub-atmospheric pressure in the range from 30 mm Hg to 60 mm Hg. Also, it is highly desirable to maintain the sub-atmospheric pressure whilst the binder sets, or is set, since otherwise the green foam may begin to collapse.

The present method has the further advantage that the foam density and the pore distribution in the foam may be readily controlled by, for example, one or more of the following: varying the viscosity of the binder, varying the particle size of the refractory material in step (i); varying the length of time of the step (ii) treatment; using a diluent in the formation of the mixture in step (i); varying the amount of gas or vapour entrapped in step (i) by, for example, control of agitation of the mixture; and heating the mixture produced in step (i). Also, the present method has the still further advantage that it may readily be scaled up.

We prefer that, in the present method, the final foamed, refractory material is prepared in the foam of an artefact.

The invention also provides a foamed, inorganic refractory material prepared by the method of the present invention.

The invention will now be particularly described in the following examples.

EXAMPLE 1

5 "Araldite" (Registered Trade Mark) epoxy resin was mixed with commercial silicon powder (150 g) and a curing agent added so that the weight of resin plus curing agent used was 100g. The mixture was re-mixed and heated in an oven at a temperature in the range from 90°C to 120°C. The mixture, which contained entrapped air, was then agitated by a mechanical whisk to introduce more air into the mixture.

10 The mixture was placed in a vacuum chamber and evacuation of the Chamber, by pumping, commenced when a exothermic reaction had started. A vacuum of 30 mm Hg to 60 mm Hg was maintained in the chamber and held until the resin had set. 20 The vacuum treatment caused the mixture to foam and gave rise to a green foam.

The green foam was removed from the vacuum chamber and debonded by heating at a rate of 5°C per hour up to 700°C under a vacuum of 10^{-3} to 10^{-4} mm Hg in a vacuum furnace. The foam was then transferred to a nitriding furnace containing an atmosphere of nitrogen and heated according to the following schedule

30	Temperature (°C)	Time (hours)
	Ambient to 1100	11
	1100	1
	1100—1150	10
	1150	30
35	1150—1300	10
	1300	10
	1300—1350	16
	1350	20
40	1350—1450	20
	1450	8

The product was foamed silicon nitride having a density of 0.6 g/cc.

EXAMPLE 2

45 The procedure of Example 1 was repeated with the exception that 200 g of silicon powder was used and 20 g of an "Araldite" (Registered Trade Mark) diluent (identified by the trade reference DY021) was included in the initial mixture.

50 The product was foamed silicon nitride having a density of 0.7 g/cc.

EXAMPLE 3

55 The procedure of Example 1 was repeated with the exception that aluminium oxide powder (175 g) was used instead of silicon powder and that the firing was carried out by heating at a rate of 100°C per hour in air to 1450°C, and holding this temperature

for 4 hours. The product was foamed aluminium oxide having a density of 0.80 g/cc.

WHAT WE CLAIM IS:—

1. A method of preparing a foamed, inorganic refractory material comprising the steps of

(i) preparing a mixture of powdered inorganic refractory material, as hereinbefore defined, and a binder under conditions such that gas or vapour is entrapped within the mixture;

(ii) subjecting the mixture to sub-atmospheric pressure to foam the mixture thereby to produce a green foam; and

(iii) subsequently debonding to remove the binder and firing to produce the final foamed, inorganic refractory material.

2. A method according to claim 1 wherein, in step (i), the refractory material and the binder are mixed and then mechanically agitated in air so that the air is entrapped within the mixture.

3. A method according to either of the preceding claims wherein the refractory material in step (i) is silicon and the firing is carried out in a nitriding atmosphere to convert the silicon to silicon nitride which constitutes the final refractory material.

4. A method according to claim 1 or to claim 2 wherein the refractory material in step (i) and in the final material is alumina.

5. A method according to any of the preceding claims wherein the sub-atmospheric pressure is in the range from 30 mm Hg to 60 mm Hg.

6. A method according to any of the preceding claims wherein, in step (ii), the sub-atmospheric pressure is maintained whilst the binder sets, or is set.

7. A method according to any of the preceding claims wherein a diluent is used in the formation of the mixture of the refractory material and the binder in step (i).

8. A method according to any of the preceding claims wherein the final refractory material is prepared in the form of an artefact.

9. A method of preparing a foamed, inorganic refractory material substantially as described herein with reference to any one of the examples.

10. A foamed, inorganic refractory material prepared by a method according to any of the preceding claims.

K. R. MANSELL,
Chartered Patent Agent,
Agent for the Applicants.